DOE/NASA/51040-35
NASA TM-82765

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Under Interagency Agreement DE-AI01-77CS51040

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ABSTRACT

The feasibility of using ultrasonic velocity as a measure of bulk density of sintered alpha silicon carbide was investigated. The material studied was either in the as-sintered condition or hot isostatically pressed in the temperature range from 1850° to 2050° C. Densities varied from approximately 2.8 to 3.2 g/cm^3. Results show that the bulk, nominal density of structural-grade silicon carbide articles can be estimated from ultrasonic velocity measurements to within 1 percent using 20 MHz longitudinal waves and a commercially available ultrasonic time intervalometer. The ultrasonic velocity measurement technique shows promise for screening out material with unacceptably low density levels.

INTRODUCTION

Reliable performance of advanced automotive gas turbines depends on the ability to assure that components placed in service meet the specifications assumed in design and life prediction analyses. Reliability assurance requires the availability of nondestructive evaluation (NDE) techniques not only for defect detection, but also for verification of physical and mechanical properties. In the absence of overiding factors (for example, large single pores or surface cracks) the strength of structural ceramics can generally be expected to vary as a function of density (ref. 1). Because current cost-effective fabrication techniques produce ceramic parts that normally are not fully dense, it is essential to have an NDE method that can identify parts having unacceptably low levels of density.

The velocity of ultrasonic waves through a solid is related to the elastic constants of the material (ref. 2). For longitudinal waves:

$$v_\ell = \left[\frac{E}{\rho}\,\frac{1-\mu}{(1+\mu)(1-2\mu)}\right]^{1/2} = \left(\frac{M}{\rho}\right)^{1/2}$$

where E is the elastic modulus, ρ is the density, and μ is Poissons' ratio. M can be simply referred to as the longitudinal modulus. Since the velocity of sound in a solid medium is much higher than in a gaseous medium, the average velocity in a porous material can be expected to increase with increasing density, rather than decrease as the above equation implies. (This of course requires that the derivative of M with respect to density be greater than unity.) Indeed, previous studies have shown that a direct

*Work performed for U.S. Department of Energy under Interagency Agreement DEAI01-77CS51040.

relationship does exist between ultrasonic velocity and bulk density of sintered powder metal parts (ref. 3) and a silicon carbide material (ref. 4). The latter investigation was performed on a type of silicon carbide used to make dielectric materials with densities ranging from about 1.6 to 2.2 g/cm^3.

The purpose of this paper is to show that the direct relation between ultrasonic velocity and density noted above holds for structural silicon carbide with densities in the range from approximately 2.8 to 3.2 g/cm^3 (88 to 99 percent theoretical) and that ultrasonic measurements can be used to predict or verify density. Consideration is given to degree of accuracy of density prediction and the effect of various microstructural factors on ultrasonic velocity.

MATERIAL AND TEST PROCEDURE

Specimens of sintered alpha silicon carbide with densities ranging from about 2.83 to 3.18 g/cm^3 were utilized in this investigation. The specimen dimensions and material conditions are given in table 1. Ceramic plates were procured in three separate batches during a time frame of about three years. The material was initially in the form of square plates measuring 5 by 5 by 0.64 cm, in the as-sintered condition. A total of 15 plates were investigated. The seven plates from batch A were sliced into two equal parts. One half of each plate was hot isostatically pressed (HIPed) at one of three temperatures, 1850°, 1950°, or 2050° C, for two hours at 138 MPa (20 ksi). Both halves were subsequently machined into rectangular specimens measuring 3.2 by 6.4 by 25 mm. The six plates in batch B were HIPed in whole at either 1950° or 2050° C and then machined into specimens with dimensions similar to batch A bars. The remaining two plates, from batch C, were HIPed at 2050° C but were otherwise not altered. A total of 195 test specimens were produced, of which 152 were HIPed while the rest remained in the as sintered condition. Densities were calculated from bulk weight and volume measurements of the test specimens.

Velocity Measurements

The determination of ultrasonic velocity involves measurement of time, t, and distance, d, where velocity is given by $v = d/t$. Here, d is defined as four times the thickness of the test specimen and t represents the time for a sound wave to make two round trips through the thickness. The specimen thickness, nominally either 3.2 mm or 6.4 mm, was measured to the nearest 2.5 μm.

A diagram of the pulse-echo overlap method for measuring time of travel is shown in figure 1. The heart of the system is the ultrasonic pulser-receiver used to drive the piezoelectric element in the transducer. The module also amplifies the return signals and provides an intensified sweep that highlights the signals of interest, while disregarding all others. An oscillator is used to drive a horizontal amplifier of a dual trace oscilloscope and provide for overlap of the intensified signals on the CRT. Overlap occurs when the oscillator frequency is equal to the reciprocal of the time interval between the intensified signals. A digital counter is used to read out either the oscillator frequency or the time of travel directly.

All components of the system are available commercially. An analysis of the accuracy of the pulse-echo overlap technique is given in reference 5. An error analysis indicated that specimen thickness measurement may be a greater source of error than measurement of time of travel.

A broadband longitudinal wave transducer with a 6.4 mm diameter active element and center frequency of 20 MHz was used for the velocity measurements. The active element was bonded to a fused silica delay buffer to enhance echo definition. Glycerine couplant was used between specimen and buffer. The silicon carbide studied exhibited low ultrasonic attenuation, and recovery of high frequency, short wave length signals was no problem even in the lowest density samples.

Velocity measurements were made at three locations along the length of the 25 mm rectangular test bars. The readings were averaged and plotted against the average density of the sample. For the two square uncut plates velocity was measured in the center of each quadrant, averaged, and then plotted against the average density of the plate.

RESULTS

Plots showing the observed relations between ultrasonic velocity and density of alpha silicon carbide from batch A are shown in figures 2 to 4. Figure 2 contains the data taken from specimens machined from two plates. Half the specimens from each plate represent the material in the as-sintered state while the other half represent the material after hot isostatic pressing (HIPing) at 1850° C. The data show that the variation in density within each plate is about one percent of nominal in both the as-sintered and HIPed conditions. However, the average increase in density due to HIPing is only about 0.25 percent and is reflected by a minor change in ultrasonic velocity. Note that the density is plotted on an expanded scale to show that the data for each plate are separate and distinct from each other. The differences do not appear to be significant. Figure 3 contains plots of data from specimens of two plates in both the as-sintered condition and HIPed at 1950° C. The velocity and density of one plate is significantly higher than for the other in the as-sintered condition. However, after HIPing, the velocity and density are similar. This suggests that the higher initial density of the first plate is very nearly the maximum attainable by HIPing at 1950° C. Figure 4 shows the results of HIPing at 2050° C on the density and corresponding ultrasonic velocity of the remaining plates from batch A. The densities of these plates in the as-sintered condition were relatively low. After HIPing at 2050° C however, the densities and velocities of all three plates increased to the highest values observed in the set of plates from batch A. Perhaps even more significant is the reduced density variation and close grouping of the data points after HIPing relative to the as-sintered material. Finally, figure 5 presents the data obtained from batch B. All of the data from this batch of material is for the HIPed condition at temperatures of 1950° and 2050° C. No data is available in the as-sintered condition. Two points can be made with respect to this curve; (1) the densities and velocities observed for the material HIPed at 2050° C are higher than for material HIPed at 1950° C, and (2) not all of the material HIPed at 2050° C densified to the same degree. The plot snows that the density of the large grained material did not increase as much as the

smaller grained material. Also, the ultrasonic velocities of the large grained specimens do not fall on the same curve as the rest of the data from batch B. The maximum deviation in terms of density however, is only of the order of one percent.

Figure 6 is a composite plot which contains all the extreme values in terms of velocity and density from the thirteen plates represented in figures 2 to 5 (i.e., only the highest and lowest values of velocity and density in each plate are plotted) to illustrate the degree of scatter while minimizing the number of plotted data points. The four data points in the lower half of the figure are additional data obtained from the two plates of batch C, before and after HIPing. The latter plates were specially processed by Carborundum Company and are not a standard product. The two points at the lower left of the figure show that no measurable densification was accomplished by HIPing of the plate with initial density of 2.83 g/cm^3, while the ultrasonic velocity increased only slightly. The other plate with initial density of about 2.92 g/cm^3 was increased to about 2.99 g/cm^3 by HIPing with a corresponding velocity increase.

DISCUSSION

It is evident from the results that a direct functional relationship exists between ultrasonic velocity and bulk density of sintered alpha silicon carbide. The data scatter band in figure 6 indicates that measurement of the velocity of longitudinal ultrasonic waves at a frequency of 20 MHz can serve as an estimator of density to within approximately one percent. While data at the lower densities are limited compared to the higher density region, they suggest that velocity measurements can be a viable quality control tool for screening out low density components. The data of figures 2 to 4 further indicate that, given a sufficiently large number of measurements, subtle density differences of less than one percent can be revealed.

Some evidence of microstructural effects other than density was observed in two of the fifteen silicon carbide plates utilized in this investigation. Figure 5 shows that ultrasonic velocities for large grain specimens were slightly higher than might be expected relative to the rest of the data. Photomicrographs in figure 7 show comparative microstructures for large and small grain material. It is apparent that the grain size in (fig. 7(a)) is much greater than in (fig. 7(b)). Many of the grains in the large grained material are also elongated or rod-like rather than equiaxed. In addition, the voids are larger and more widely spaced in the large grained specimen. Cumulatively, these microstructural features appear to affect ultrasonic velocity. Whether the character of the grains is more important than the character of the voids has not been determined conclusively. It appears, however, that grain morphology has a lesser effect than porosity or pore morphology. This is inferred because of the strong relation observed in this investigation, as shown in figure 6. This strong velocity-density relationship holds over a wide variety of material conditions. This study indicates that ultrasonic velocity measurement is viable for estimating bulk density of structural ceramic materials provided calibration curves are generated for the particular materials/structures of interest. Even in lieu of calibration curves, qualitative ranking according to density can still be obtained.

SUMMARY AND CONCLUSIONS

This paper describes correlations found between ultrasonic velocity and material density for a series of SiC specimens both in the as-sintered and hot isostatically pressed conditions, and over a density range of approximately 2.8 to 3.2 g/cm^3. Ultrasonic longitudinal-wave velocity measurements can be a useful tool for estimating bulk density of ceramics suitable for turbine engine components. Given a calibration curve for a particular material, ultrasonic estimates of density to within a percent of true bulk density are possible, and articles having unacceptably low density levels can be identified. If a calibration is not available, velocity can still be used to qualitatively rank the materials on the basis of density.

Recommendations for future work include (1) applying the ultrasonic velocity technique to the measurement of density in actual parts, and (2) conndducting a detailed study on the interaction between microstructure and ultrasonic measurements with emphasis on void/pore morphology.

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TABLE 1. - ALPHA SILICON CARBIDE TEST SPECIMENS

Batch	Condition	Dimensions, cm
A	As sintered and ground	0.32 by 0.64 by 2.5
A	HIPed 1850° C and ground	↓
A	HIPed 1950° C and ground	↓
A	HIPed 2050° C and ground	↓
B	HIPed 1950° C and ground	↓
B	HIPed 2050° C and ground	↓
C	As sintered	5 by 5 by 0.64
C	HIPed 2050° C	5 by 5 by 0.64

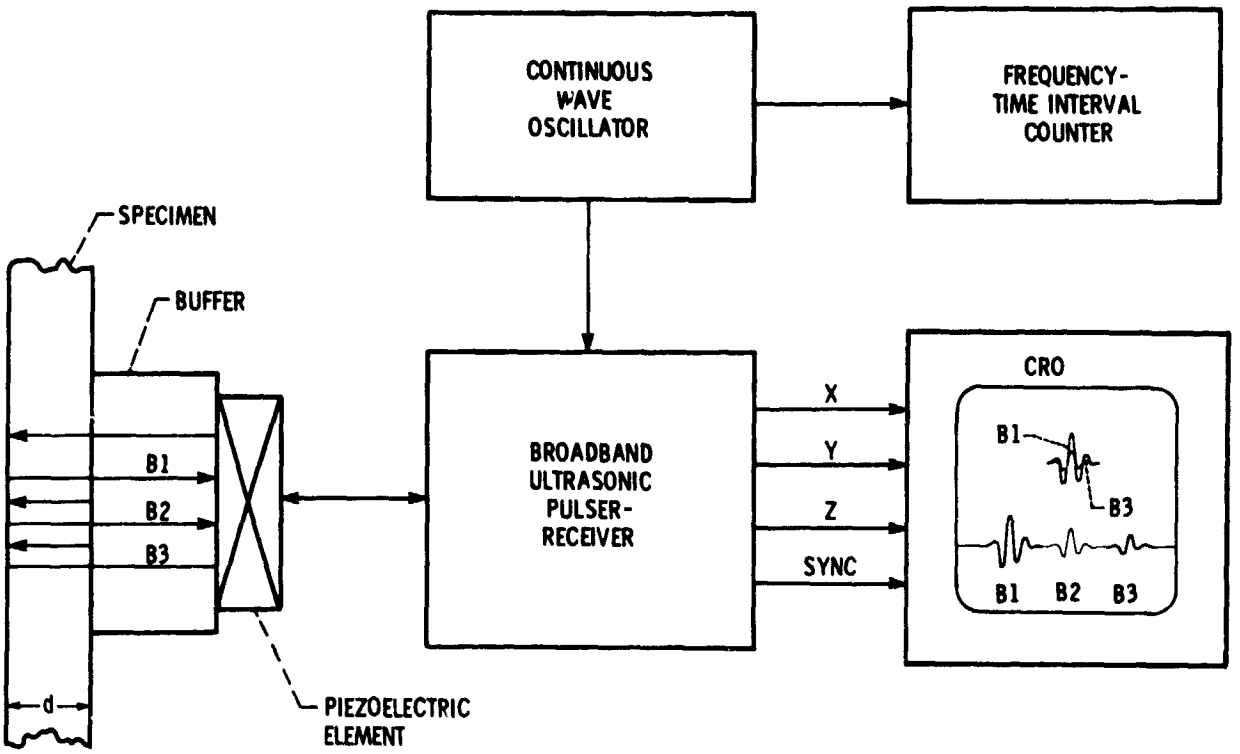


Figure 1. - Pulse-echo overlap system of measuring time of travel of ultrasonic waves.

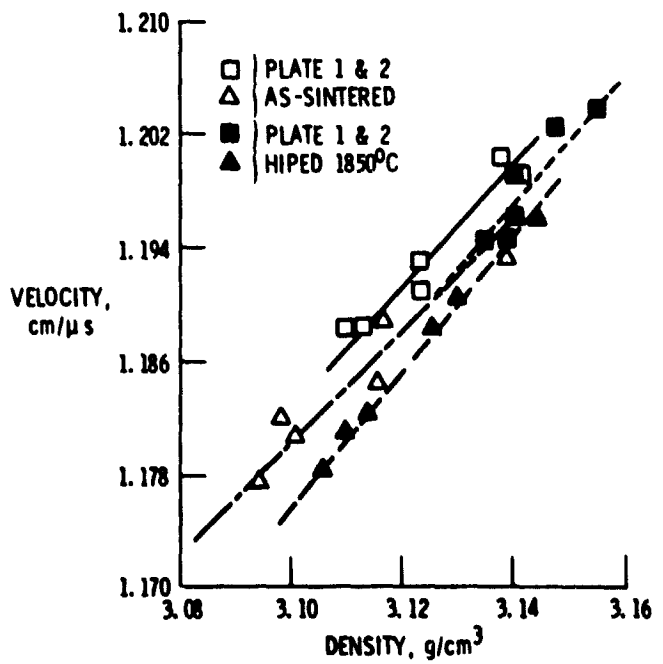


Figure 2. - Effect of hot isostatic pressing (HIPing) at 1850° C on ultrasonic velocity and density of sintered αSiC.

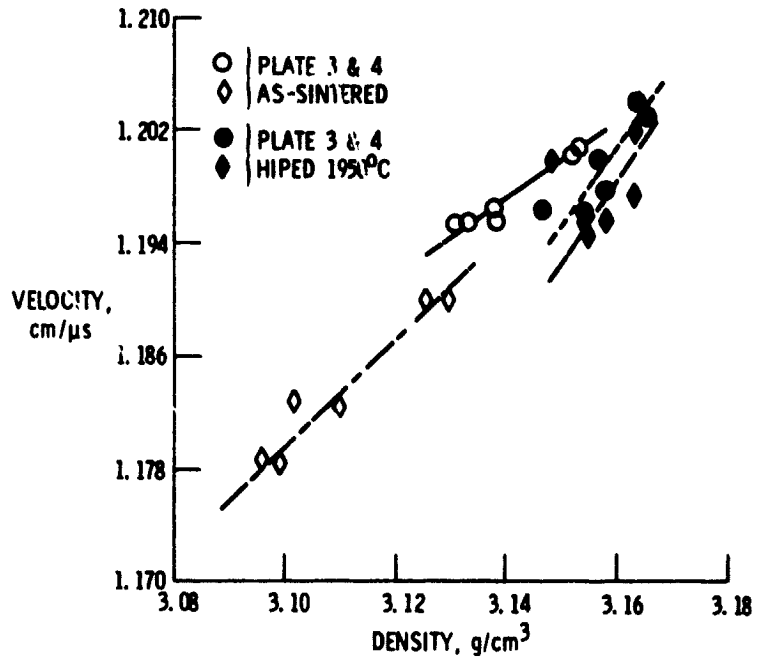


Figure 3. - Effect of hot isostatic pressing (HIPing) at 1950° C on ultrasonic velocity and density of sintered α SiC.

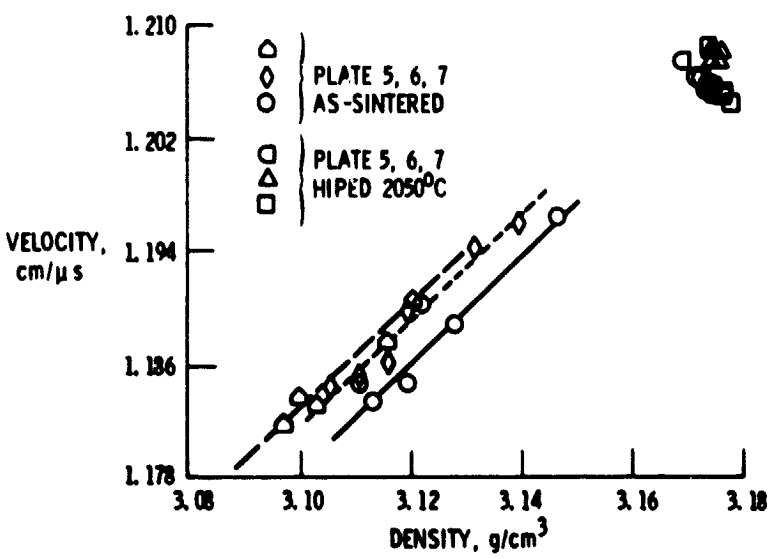


Figure 4. - Effect of hot isostatic pressing (HIPing) at 2050° C on ultrasonic velocity and density of sintered α SiC.

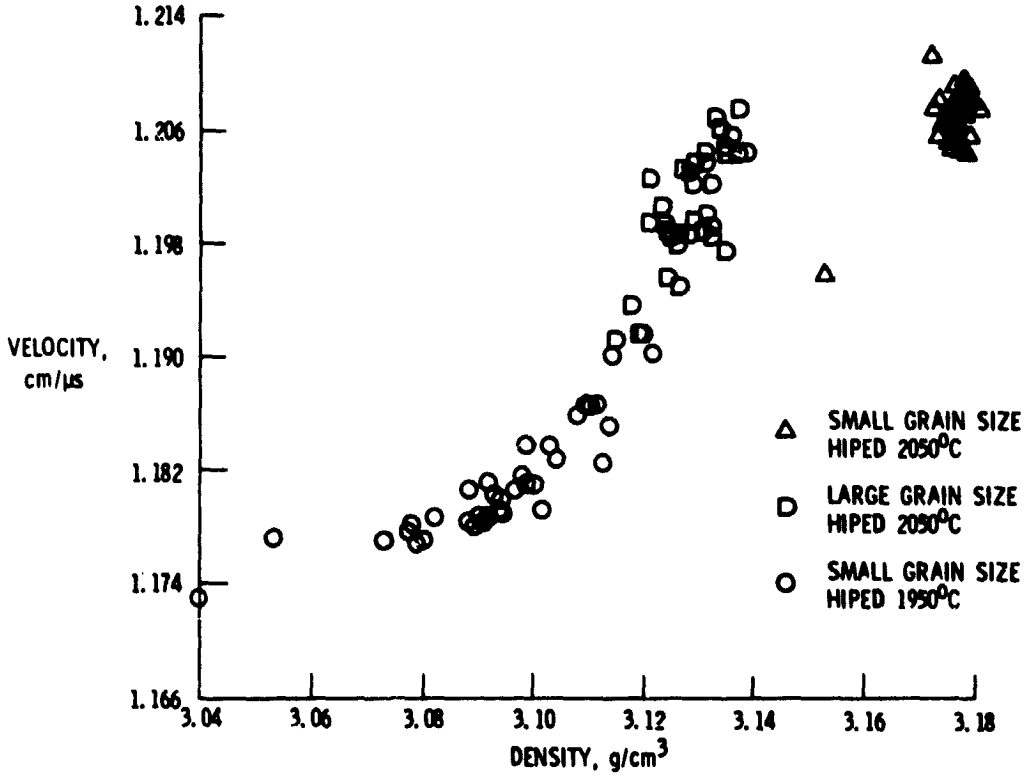


Figure 5. - Effect of density on ultrasonic velocity of hot isostatically pressed (HIPed) αSiC. Batch B material.

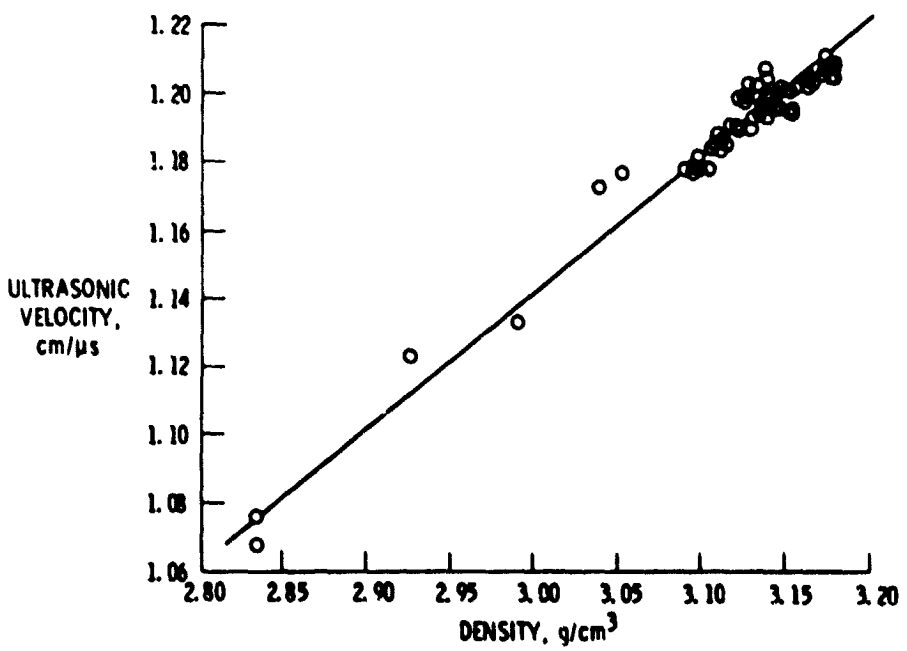
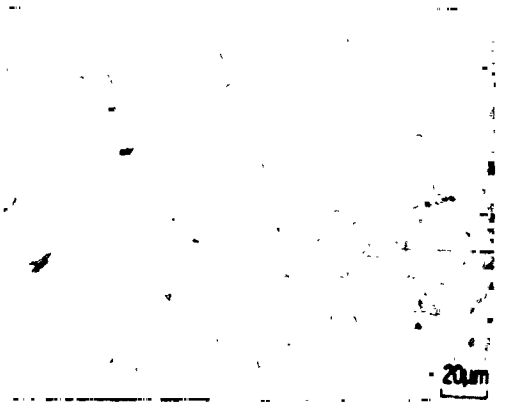


Figure 6. - Composite curve showing velocity-density relation for all material conditions represented in this program.

(a) Large grain size material.



(b) Small grain size material.

Figure 7. - Microstructure of sintered alpha silicon carbide showing grain size and void distribution in two plates. Both plates were hot isostatically pressed at 2050° C.